Metastabilty Driven Hierarchical Microstructural Engineering: Overview of Mechanical Properties of Metastable Complex Concentrated Alloys

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Abstract

Complex concentrated alloys (CCAs) extend the compositional paradigm shift of high entropy alloys (HEAs) to new microstructural opportunities. CCAs provide opportunities for tunable performance by manipulating deformation mechanisms. Fe-Mn-Co-Cr-Si alloys exhibit potential for a combination of phase transformation and twinning. These alloys give greater flexibility for tailoring transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP), which have guided design of next-generation steel alloys over the last 20 years to a new level. For TRIP CCAs, the ductility can be extended to as high as 50% while maintaining a strength exceeding 1 GPa. The Fe-Mn-Co-Cr-Si alloys show extensive gamma (f.c.c.) to epsilon (h.c.p.) phase transformation followed by additional twinning in the epsilon phase. Combination of ultrafine grained microstructure and crack tip transformation leads to enhanced fatigue limit in TRIP CCAs. Design of non-equiatomic CCAs provides a vast, and vastly unexplored compositional space for developing new alloys with tunable properties.

Keywords: Complex concentrated alloys; high entropy alloys; transformation; twinning; c/a ratio

1. Introduction

High entropy alloys (HEAs) have attracted attention of researchers in the last decade due to scientific interest in understanding their microstructure-mechanical property relationships. The central theme of discovery of first two compositions by Yeh *et al.* [3] and Cantor *et al.* [4] was formation of single phase solid solution alloy by adding together equimolar amounts of transition elements, namely Fe, Mn, Co, Cr, Ni. These alloys changed the paradigm of defining solvent and solute in solid solution alloys. The design of these HEAs was based on the concept that, increasing the configurational entropy of the alloy system by maximizing the constituent

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elements would lead to higher driving force for solid solution formation over intermetallic compounds at higher temperature [1-2].

Design of equiatomic HEAs moved the alloy search space from the corners of the hyperdimentional compositional space to the center, thereby focusing on the unexplored region of the space [1-2]. The framework for discussion of properties in these alloys started referring to four core principles: (1) high entropy effect, (2) lattice distortion effect, (3) sluggish diffusion effect, and (4) cocktail effect. Mishra et al. [5] focused on the lattice distortion effect in discussing the mechanical behavior of these alloys. Note that the simple solid solution alloys only have (a) solid solution strengthening and (b) grain boundary strengthening in the conventional strengthening mechanisms framework. The impact of these alloying approach on stacking fault energy (SFE) is important to quantify for discussion of mechanical properties. Lowering the stacking fault energy of face centered cubic (f.c.c.) phase below a critical value (around 30 mJ/m^2) triggers additional strengthening mechanism in the form of deformation induced twinning. Along with twinning, formation of multiphase microstructure was also attempted by the design of very popular Al$_x$CoCrFeNi HEAs. From this category of HEAs, Al$_{0.1}$CoCrFeNi HEA was studied in detail by various research groups which concluded that, twinning induced plasticity dominated the room temperature deformation in this alloy along with the exceptional failure resistance at cryogenic temperatures [6-8]. Along the similar approach, Raabe and co-workers [9-10] have expanded this alloying approach to non-equiatomic HEA design mainly targeting formation of f.c.c. and h.c.p. phases in the microstructure which they termed metastable dual phase HEAs. The onset of metastable dual-phase alloys is an interesting research line because it enables creation of nanointerfaced microstructures as envisioned by Lu *et al.* [11] for enhanced strength-ductility combination. The journey of unconventional alloy design approach from HEA to multi-principal element alloys (MPEAs) to complex concentrated alloys (CCAs) is discussed briefly in the next section.

2. Morphing towards a broader approach for complex concentrated alloys

While the HEA design approach is transformative and takes the solvent-solute alloying paradigm to multi-principal elements, the equiatomic approach is very limiting (Fig. 1). The approach can be expanded from the equiatomic center point of the compositional search space to non-equiatomic compositions. Aligned with this is the shift from single phase solution alloy to multi-phase multi-principal element alloy with multiple strengthening mechanisms (Fig. 1).

There are three distinct considerations that can be added beyond the solid solution strengthening: (a) lowering the stacking fault energy to promote dissociation of full dislocations into partials, (b) formation of second phases to add precipitation strengthening, and (c) interfacial strengthening by promoting grain refinement, formation of deformation-induced twins and deformation-induced transformation. By selectively choosing minor elements in addition to major constituent elements, non-equimolar complex concentrated alloys (CCAs) can provide opportunities to tailor microstructure to trigger various mechanisms at different stages of deformation. In this short overview, the CCAs are divided in two categories, (a) compositions that promote twinning (TWIP CCAs), and (b) compositions that promote transformation (TRIP CCAs). Only examples of strength-ductility variation and fatigue life of TRIP CCAs are highlighted. The terms HEAs and CCAs are used interchangeably in this paper [1-12].
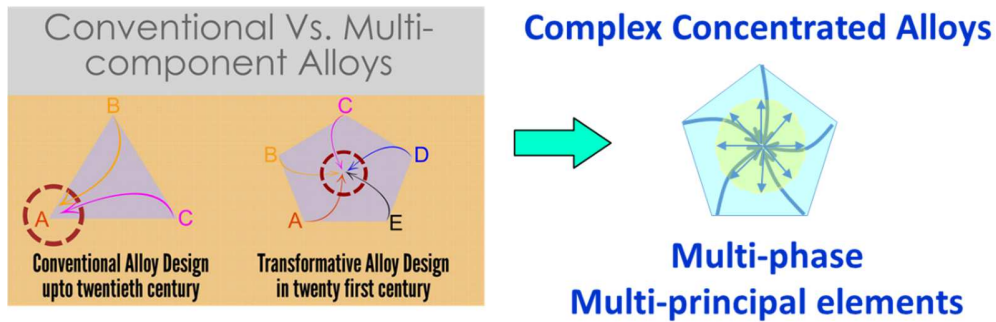

Fig. 1: Schematic representing the shift in the alloy design strategy from conventional to HEAs and now to CCAs.

3. Metastable complex concentrated alloys

3.1. Advent of a transformative alloy

A new class of non equiatomic CCA was designed by Li *et al.* [9], which they referred as dual phase transformation induced plasticity $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ HEA (DP-HEA). They showed extreme work hardening ability for DP-HEA due to γ-f.c.c.→ ε-h.c.p. transformation during strain as a result of increased metastability of γ matrix and thus resulted in a strength and ductility of 850 MPa and 70% at room temperature. The only limitation of the alloy was poor yield strength (~290 MPa) which they tried to improve by doing either severe plastic deformation like friction stir processing (YS= 400 MPa), [10] or minor addition of carbon (0.5 at.%) in DP-TRIP HEA (YS=500 MPa, [12]).

3.2. Metastability design for dual phase alloys: Linkage to stacking fault energy

Li *et al.* [9] reported that ε martensite phase is harder than the γ phase in TRIP HEAs and ε phase fraction prior to deformation along with grain size influence the strength-ductility synergy in this alloy. Thus, apart from the grain size, if the fraction of harder ε phase is increased more than 50 %, then it would push the YS of TRIP HEAs to higher values than that obtained by Li *et al.* [9]. To obtain such a large ε fraction in the TRIP-HEA, the $\Delta G^{\gamma \to \varepsilon}$ should be increased by proper selection of constituent elements. From the TRIP steel literature, we know that Si and Cr are ε phase stabilizers whereas Mn and Ni are γ austenite stabilizers [13]. On a similar note, we did thermodynamic simulations by considering addition of Si in DP-TRIP HEA system by increasing the Cr level and decreasing the Mn content The effect of Si addition on the RT $\Delta G^{\gamma \to \varepsilon}$ value was evaluated using ThermoCalc TCHEA2 database as shown in Fig. 2 (a). The more negative value of $\Delta G^{\gamma \to \varepsilon}$ with increasing Si content in comparison with $\Delta G^{\gamma \to \varepsilon}$ of the alloy having no Si indicates increased tendency towards ε formation. This increased tendency of ε formation at RT in turn reflects increased γ metastability by the addition of Si in DP-HEA matrix. Accordingly we designed new TRIP HEA/CCA compositions with 5 at.% Si addition thereby resulting to a composition of $Fe_{42}Mn_{28}Co_{10}Cr_{15} Si_5$. We termed this new $Fe_{42}Mn_{28}Co_{10}Cr_{15} Si_5$ HEA as DP-5Si-HEA [14]. The physical significance of Si addition towards ε stability is by virtue of decreased SFE (γ_{SFE}) of the γ phase. The γ_{SFE} of the matrix phase is related with the $\Delta G^{\gamma \to \varepsilon}$ by,

$$\gamma_{SFE} = n\rho\Delta G^{\gamma \to \varepsilon} + n\sigma^{\gamma \to \varepsilon} \tag{1}$$

where γ_{SFE}, n, ρ and $\sigma^{\gamma \to \varepsilon}$ represent stacking fault energy, the number of planes, the planar density and the interfacial energy between the two phases, respectively. Thus, as the $\Delta G^{\gamma \to \varepsilon}$ becomes more negative, γ_{SFE} starts decreasing correspondingly and hence increases metastability of the matrix phase [10-15]. Physically, it means that intrinsic stacking faults (ISFs) are stabilized in the microstructure in higher number density which are considered to be the nucleation sites for ε-phase formation. In short, decreased $\Delta G^{\gamma \to \varepsilon}$ is helpful in increasing the probability of stable ISFs in the microstructure which favors the easy stabilization of ε-martensite [10-15]. Based on this principle we designed extremely metastable TRIP HEAs having compositions mentioned in Table 1 and engineered its microstructure with friction stir processing (FSP). From ThermoCalc simulations with TCHEA2 database, the 5% Si containing

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HEAs showed more negative values (-750 J/mol) for $\Delta G^{\gamma \to \varepsilon}$ than the original DP-TRIP HEA without Si (420 J/mol), which supports the fact that Si addition made the γ matrix extremely metastable (Fig. 2 (a)).

Recently, Liu et al. [15] demonstrated that, Co addition also increases the γ matrix metastability in comparison with Ni and Cr addition in Fe-Mn-Co-Cr matrix. So, for increasing γ metastability even further, we modified the DP-5Si-HEA composition by increasing the Co content to 20% from 10 % at the expense of Mn content thereby resulting to a composition of $Fe_{40}Mn_{20}Co_{20}Cr_{15}Si_5$ (all in at. %). We termed this new $Fe_{40}Mn_{20}Co_{20}Cr_{15}Si_5$ HEA as CS-HEA.
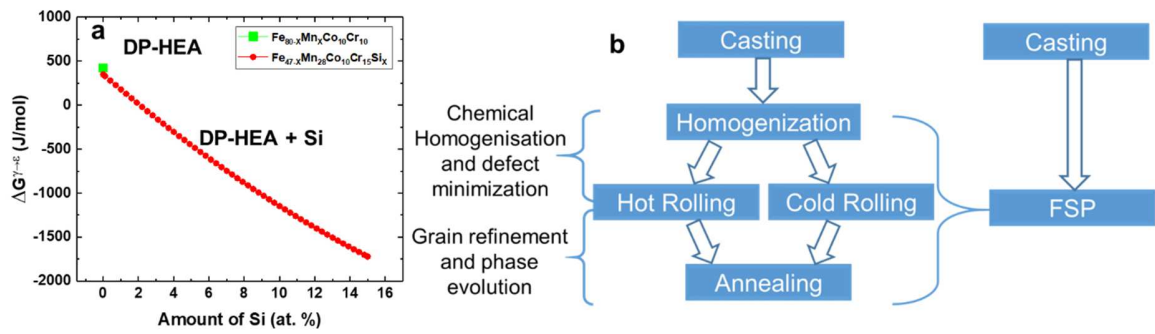

Fig. 2: (a) ThermoCalc predictions for $\Delta G^{\gamma \to \varepsilon}$ at room temperature for HEA with and without Si using TCHEA2 database, and (b) processing paths used for metastable HEAs to evaluate microstructural dependence.

All the HEAs were produced by vacuum arc-casting in a cold-copper crucible. The vacuum level achieved was approximately 300 µm, and the chamber was backfilled with argon to 1 atm prior to each melt, using pure metals and ingot dimensions of $300\times100\times6$ mm^3. FSP was selected as a primary processing route due to its unique nature of imparting severe plastic deformation at higher temperature. The shear driven transport of elements chemically homogenizes the microstructure after FSP thereby eliminating the need for any separate homogenization [10, 14] treatment before processing (Fig. 2 (b)). Moreover, ε formation being sensitive to imposed strain and temperature, simultaneous impartment of these provides opportunities to obtain many different variants of the ε dominated microstructure at room temperature.

4. Microstructure and mechanical properties of metastable alloys

Figs. 3 (a-d) summarizes some examples of microstructural evolution for a few of newly designed metastable TRIP HEAs, namely, DP-5Si-HEA [14] and CS-HEA [16] in comparison with the DP-HEA [10] and DP-iHEA [17] under similar FSP conditions. It is quite clear that Si

addition completely changed the microstructural stability thereby resulting in >80 % ε martensite phase (Figs. 3(c,d)) at room temperature which was not feasible to attain in TRIP steels or HEAs earlier. Figs. 3 (a-d) also supports the thermodynamic predictions made in section 3 and suggest that CS-HEA and DP-5Si-HEA should have very high driving force ($\Delta G^{\gamma \to \varepsilon}$) at room temperature and hence showed very high ε fraction. Moreover, Fig. 3 (e) correlates the fraction of ε phase stabilized in each HEA with the average grain size attained. It was claimed by Li *et al.* [9] and Nene *et al.* [10] that, grain size and ε fraction governs the propensity of TRIP effect in metastable HEAs. With addition of Si, it is seen that, grain size can be refined to submicron level very easily with higher fraction of ε phase formed under similar FSP processing condition. Thus, Fig. 3 (e) highlights that an uninvestigated ε fraction-grain size domain was explored due to design of Si containing metastable HEAs processed with FSP.
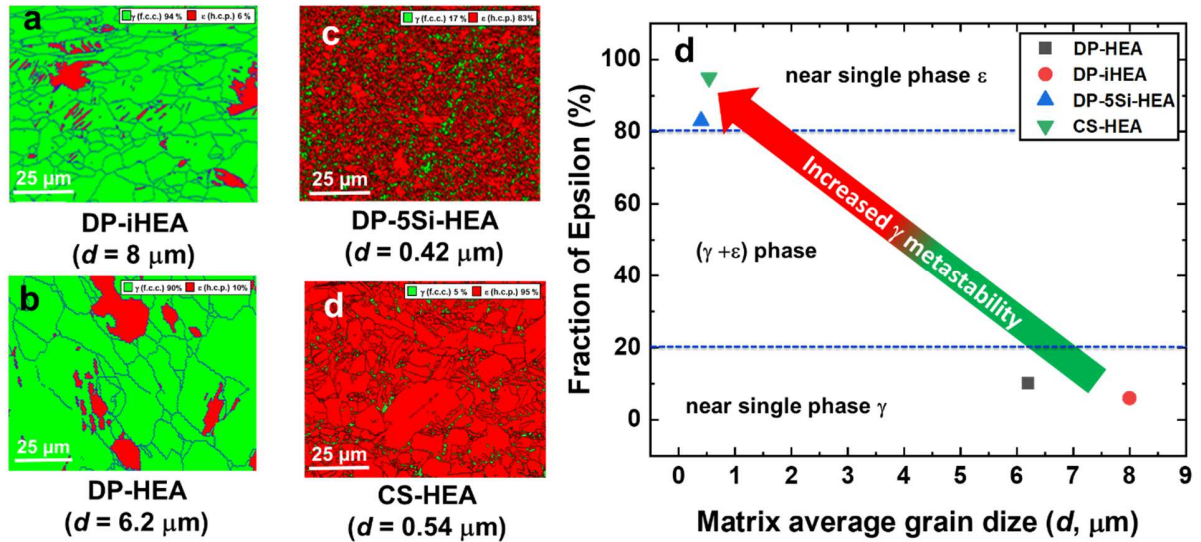

Fig. 3: (a-d) Microstructural evolution in metastable HEAs with addition of Si under similar FSP condition (350 RPM), (e) fraction of ε phase stabilized vs matrix average grain size for all HEAs under similar FSP condition.

Fig. 4 (a) display the engineering stress-strain curves for all HEAs after FSP at 350 rotations per minute (RPM). It is evident that, the ε-h.c.p. dominated HEAs showed higher yield strength (YS) than γ rich HEAs with comparable strength-ductility synergy. Among all HEAs, CS-HEA showed very high strength-ductility in spite of having very high YS compared to DP-HEA [9-13]. It is also important to note that, CS-HEA is ε-h.c.p. rich and provides a very different

response from conventional h.c.p. alloys. Conventional h.c.p. alloys are often associated with limited RT ductility due to restricted active slip systems if the c/a ratio is close to the ideal (1.633). However, CS-HEA displayed very high ductility which is an indication of activation of multiple slip activity in the material. Li *et al.* [9] proposed that prior ε phase fraction and grain size are important factors in deciding the deformation mechanisms and dominance of ε with finer grain size limits the TRIP effect in HEAs. Our results shows that though the TRIP effect is lower or missing in ε dominated HEAs (depending on the starting volume fraction of ε-phase, Figs. 3 (c,d) and Fig. 4 (a)), the ability of ε-phase to deform and partition strain effectively provides similar work hardening ability to these new alloys and hence maintain the high work hardening rate and ductility [13-16]. The strain hardening behavior is distinctly different in each case which can be explained with the work hardening curves shown in Fig. 4 (b). A distinguishing feature in these curves is the change in the extent of sustained work hardening rate over a larger range of strain in each alloy, which is mainly associated with the prior ε fraction (i.e. γ matrix metastability) obtained in the microstructure (highlighted by black dotted lines in Fig. 4 (b)). This is a topic which is rich for exploring the relative role of transformation and twinning in the overall work hardening rate and plasticity in these alloys. Note that the conventional definition of twinning induced plasticity (TWIP) and transformation induced plasticity (TRIP) implies that concurrent twinning or transformation during deformation enhances plasticity. The results so far on these transformative HEAs/CCAs suggest that the intrinsic plasticity is high and we may refer these as plasticity induced transformation and plasticity induced twinning and the conventional framework of discussion can shift as we develop better quantitative relationships describing the evolution of transformation and twinning during deformation.
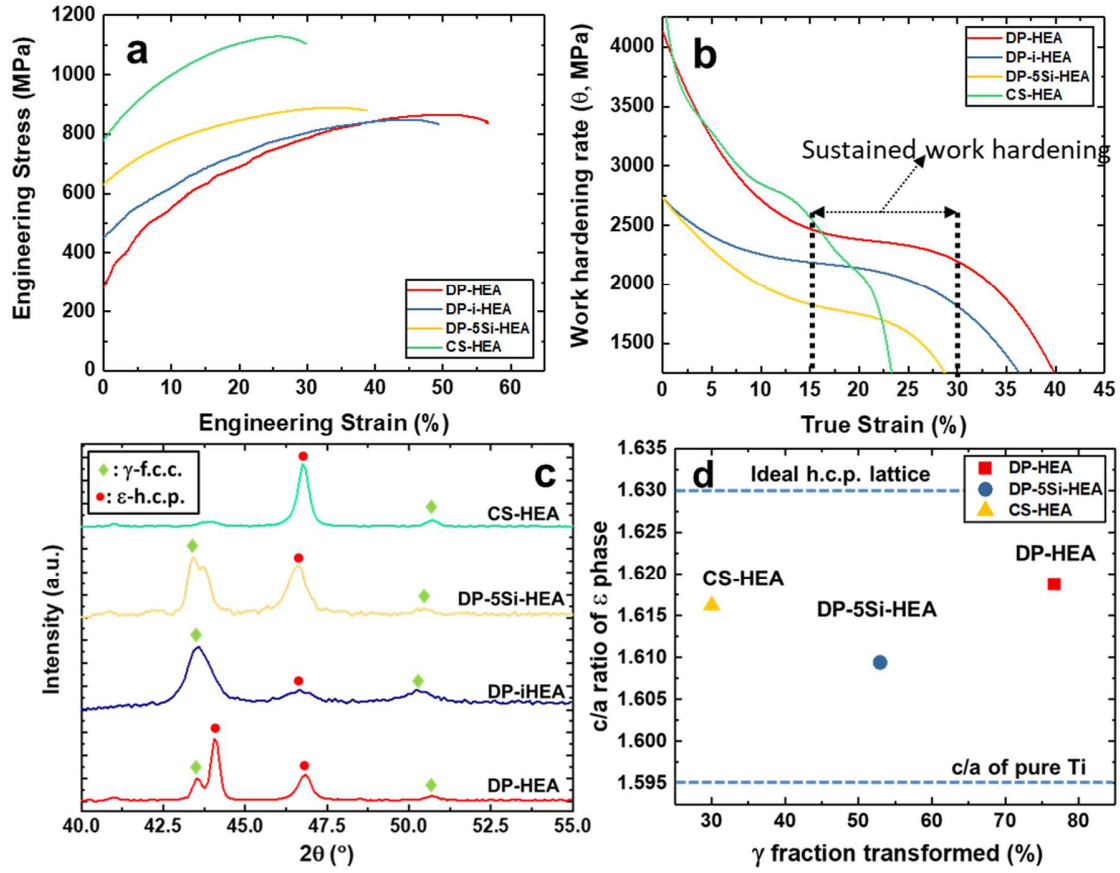
Fig. 4: (a) Engineering stress-engineering strain curves for metastable HEAs after FSP at 350 RPM, (b) work hardening behavior of metastable HEAs with and without Si addition under similar processing condition, (c) XRD patterns for all HEA, and (d) c/a ratio vs γ fraction transformed for all investigated HEAs.

Our earlier studies on the CS-HEA showed that [16-19], the ductility in ε phase is mainly attributed to the activation of multiple deformation mechanisms such as basal slip, non-basal slip activity in the early stages of deformation whereas extension twinning in the later stages. It is known that, the CRSS for the non-basal <c+a> slip can be lowered in h.c.p. materials by engineering the c/a ratio of the unit cell. As the c/a ratio drops away from the ideal ratio of 1.633, CRSS to activate non-basal slip at ambient temperature start dropping and hence the material become more ductile. Our recent results using in situ neutron diffraction validated the continuous drop in c/a ratio with deformation due to concurrent compression of c axis and expansion of a axis in the h.c.p. unit cell [19]. Fig. 4 (d) shows examples of the c/a ratio of ε phase for these

HEAs plotted with the γ fraction transformed (%) after deformation [19]. The c/a ratio was estimated using XRD as explained in our earlier work [19]. Fig. 4 (c) shows the XRD plots for all the HEAs after FSP at 350 RPM which were used to calculate c/a ratio of the ε phase. It is interesting to note that, our metastability-designed HEAs have lower c/a ratio of ε phase than ideal h.c.p. unit cell and marginally different than the c/a ratio of ε phase in DP-HEA reported by Bu *et al.* [20]. Moreover, DP-HEA showed maximum fraction transformed indicating that, most of the strain in it is accommodated by TRIP. On the other hand, CS-HEA showed lowest γ fraction transformed indicating dominance of other deformation mechanism than TRIP for strain accommodation. As a result, though both these HEAs have similar c/a values, the γ metastability influences the preferential deformation mode. This opens up further opportunities to investigate the rate of transformation and link it with the level of work hardenability [18-20]. We also found that h.c.p. dominated HEAs undergo dynamic recovery in the early stages of deformation owing to early activation of non-basal plasticity thereby resulting in the formation of nano-ε-plates. This heavily interfaced microstructure also increases the plasticity of the alloy by increases local work hardening ability as proposed by Lu *et al.* [11]. In our opinion, these results are quite different from conventional h.c.p. alloys and open up opportunities for fundamental study of deformation in these CCAs. The fine-tuning of these CCAs with addition of minor alloying elements has just started and is at a very nascent stage. The transformation is accompanied by a small volume change (the h.c.p. phase has lower volume). The compositional dependence of this volume change is unexplored at this stage and the linkage to deformation mechanism(s) has not been quantified.

Fig. 5 is a composite plot from the results of Liu et al. [21] on exceptional fatigue behavior and fatigue limit of a DP-5Si-HEA. Liu *et al.* [21] have shown than the transformative HEAs/CCAs have interesting evolution of mechanisms operating at the fatigue crack tip. Fig 5 (a_{1-4}) captures the fatigue crack nucleated in ultrafine grained DP-5Si-HEAspecimen during the high cycle fatigue testing interrupted after 121551 cycles at a reversible stress amplitude of 680 MPa [21]. The crack branching is evident along with the formation of twin like features near the main crack. Detail EBSD analysis of the region near the crack showed pronounced γ→ε transformation within the crack tip plastic zone and the KAM distribution suggested higher deformation in the ε phase field (Fig. 5 (a_4)). The crack branching seen in the Figs. 5 (b,c) is associated with the localized work hardening activity due to transformation which delays the

growth of fracture via increased mean free path for crack propagation [21]. As a result, DP-5Si-HEA showed 0.46 times UTS as a fatigue run out which is better than most of the commercial TRIP steels and HEAs [21]. Thus, this localized transformation phenomena near the crack tip gives a new strategy to improve the fatigue resistance of fine grained materials by tuning the matrix metastability which ultimately decide the extent of transformation near the crack [13-21]. As a result, this level of metastability driven alloy design can be merged with creation of hierarchal features, such as martensite plates or twins, to overcome not only the strength-ductility trade-off paradigm but also the damage tolerance of CCAs under cyclic loading.
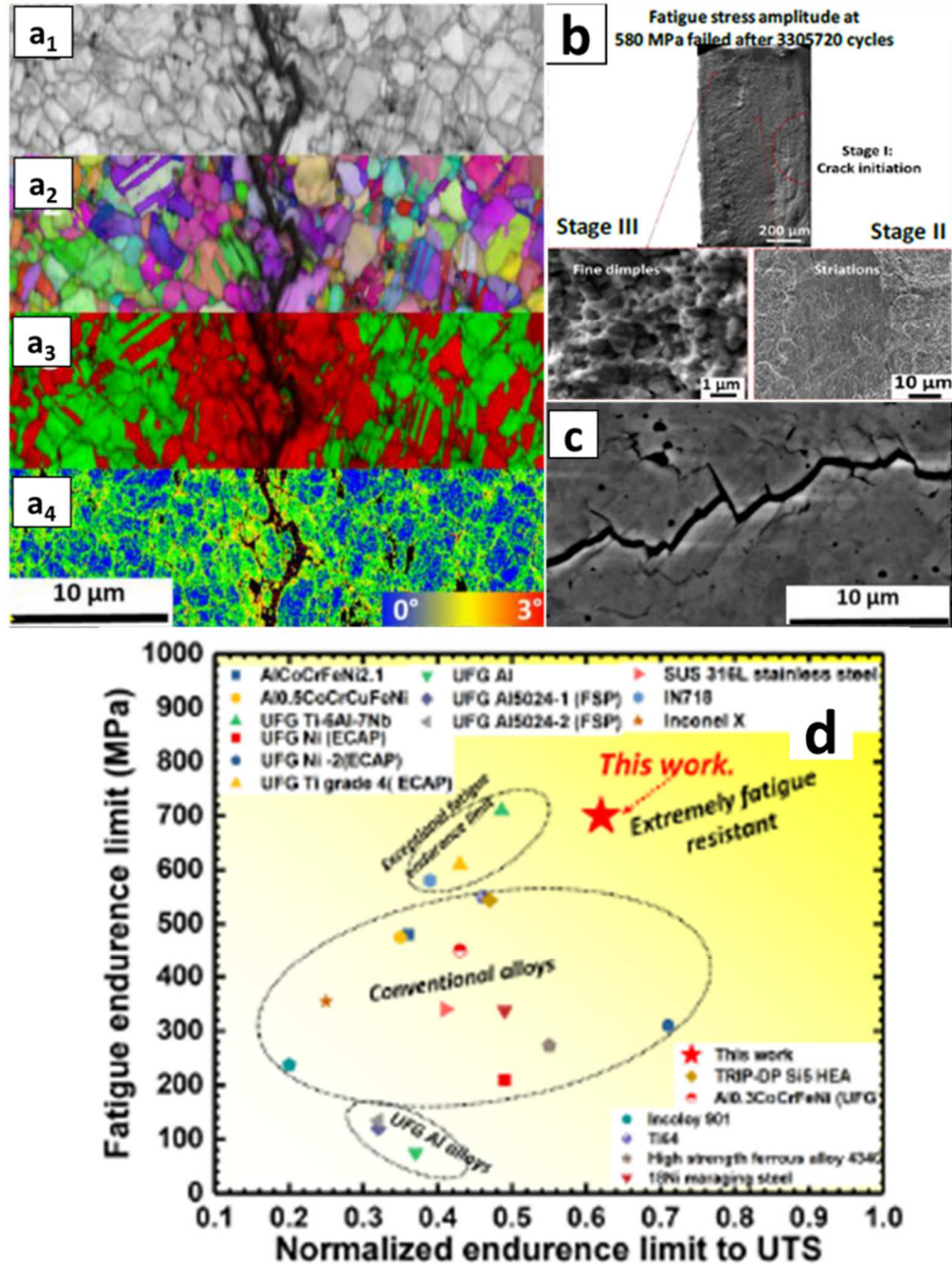
Fig. 5: ((a$_{1-4}$) Example of higher magnification scan around the crack with image quality map, IPF map, phase map, and KAM, respectively, to show the transformation around the fatigue crack, (b) fractographs of a sample tested at the cyclic stress of 580 MPa after failure at 3305720 cycles, (c) BSE image at the crack shows the fatigue crack branching, and (d) an overview plot comparing the fatigue properties of DP-5Si-HEAwith other conventional fatigue resistant alloys [21].

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5. Concluding remarks for future directions

The design of highly metastable and ε-h.c.p. dominated complex concentrated alloys provides new opportunities to explore fundamental deformation mechanisms. The microstructural engineering using high temperature severe plastic deformation through FSP extends to landscape available for exploration of mechanisms at various length scales. The possibilities of sequencing the transformation, twinning, and activation of multiple deformation mechanisms opens up opportunities to explore ultrafine grained materials in a very different than what has been possible in the past. These mechanisms are able to overcome the limitation of work hardening in ultrafine grained conventional alloys. The initial results of evolving c/a ratio of the ε phase is quite intriguing and very different from conventional h.c.p. alloys. Perhaps, these are good alloys towards the broader question of how the mechanical behavior of HEAs/CCAs are different from conventional alloys. The underlying mechanisms need to be investigated and the difference in monotonic and cyclic response provides additional opportunities. The volume change during transformation adds another dimension to the complexity in these metastable alloys.

Acknowledgments

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